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Cast Double Base Propellants: Process Mechanics

J. B. Hamann

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is by J.B. Hamann and describes the experiments used to investigate the mechanics of casting large double base booster for rockets and JATO's. Many conclusions were reached in this paper as to effects of time, temporature, pressure, on the size of cast. Particle size of the powder is also considered.  COMMENT: The result of this work will have application to many Ordnance guided missiles and Jato's. Copies are included for ORDTU, Redstone Arsenal and Picatinny Arsenal.  Copy No. 23 is for the R&D Board.  Dist by Orig: Copy to Off. Grp#1 w/o incl.

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#### EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT.

REPORT NO. 29/R/52

Cast Double Base Propellants: Process Mechanics

by

J.B. Hamann, Attached Australian Scientist.

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#### Reference: XR 430/9

#### 1. INTRODUCTION

The requirements of modern warfare for solid propellant charges for high thrust boosts and sustainers for guided missiles and for assisted-take-off units for aircraft have necessitated the production of grains of larger diameter than can be made by the extrusion of solventless cordite. Charges up to 9 inches in diameter have been produced in Great Britain from a 15 inch press and up to 10 inches in Germany from a 21\frac{3}{4} inch press; but the size of an extrusion is normally limited to an effective cross-sectional area 25 per cent of that of the press cylinder.

The increasing demand for large charges has directed attention to methods of casting propellant grains. The cast double base process depends on the ability of nitrocellulose powders to swell and coalesce into a coherent mass when treated with a suitable solvent and plasticiser. The process consists essentially of manufacturing a container, filling it with a fine-grain nitrocellulose powder, evacuating to remove volatile matter, casting with a liquid nitric ester and curing at an elevated temperature. The container is normally a cellulose acetate, convolute-wound, tube which bonds to the charge and acts as an inhibitor, whilst permitting the propellant and its bond to the restriction to be inspected.

The casting technique is not only suitable for the production of charges of large web thickness but also for those of long length and complex grain pattern.

#### 2. OBJECTS OF INVESTIGATION

Though practical developmental work has been carried out on the cast double base technique with the 6 inch pilot plant at Waltham Abbey, little knowledge has been accumulated on the theory of the 'Mechanics of Casting'. This investigation has been initiated to increase this knowledge and to provide additional data for casting long charges.

The main objects are:

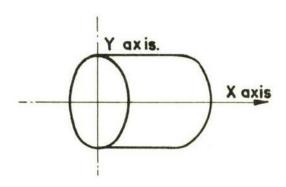
- 2.1. To measure the rate of dilatation of the casting powder and its rate of penetration by casting liquid,
- 2.2. To measure the rate of dilatation of the convolute-wound container and its rate of penetration by the casting liquid,
- 2.3. To measure the densities, and calculate the volume coefficients, of the casting powders and finished charges,
- 2.4. To determine the rate of solution of the casting powder in the casting liquid,
- 2.5. To measure the change in volume of the system on casting and during the consolidation period,
- 2.6. To determine what effect the geometrical shape, size and method of manufacture of the powder will have on the mechanics of casting,
- 2.7. To determine the optimum conditions of temperature and pressure required to east charges up to 15 feet in length.

#### 3. EXPERIMENTAL PROCEDURE

#### 3.1. Dilatation of Powder Granules

The granules of chopped cord are stuck to a thin layer of wax at the bottom of a Petri dish (Fig. 1, p.22) and covered with casting liquid; the rate of dilatation is measured at regular intervals by means of a travelling microscope.

Two grains are set in each dish, one exposing the X axis (direction of extrusion) and the other the Y axis (Fig. 2 below).



# CONVENTION FOR DIRECTION OF MEASUREMENT OF CORDITE GRAINS

FIG 2

The dish is stuck to a square of white card, the granules lined up with the cross-wires of the microscope, and a guide line marked on the card to correspond with a similar one on the microscope baseboard to ensure that the same cross-section is measured each time.

The dish and casting liquid are placed in a constant temperature and humidity cabinet (set at 20°C., 55% relative humidity) for at least 24 hours before commencing the test. The granules are then measured, immersed in casting liquid, re-measured and replaced in the humidity cabinet. Measurements are taken after 4 hours, 24 hours and daily for 4 days, after which the swollen edge is usually too indistinct for further accurate measurement. Both the outer swollen edges and the inner unaffected core of the granule are measured.

As a high percentage of the granules are not chopped cleanly by the rotary cutter during manufacture it is necessary to pick out the least distorted cylinders to obtain reproducible results.

It is also important that the granules are set truly vertically, with only one plane visible, because during swelling the sharp edge disappears and focusing becomes difficult.

The granules

The granules should be immersed in a minimum of casting liquid, to climinate as far as possible solution effects, though sufficient must be used to cover the granules completely and ensure that the swelling is three-dimensional.

Care should be exercised in handling the dishes, especially after 2 days, to prevent damage to the soft swollen edges of the granule. The stabiliser, 2-nitrodiphenylamine, is not added to the casting liquid, because its intense colour makes it impossible to follow the dilatation of the similarly coloured granules.

#### 3.2. Dilatation of Convolute-Wound Collulose Acetate Containers

Initial experiments on the dilatation of convolute-would cellulose acetate containers, when the specimens were completely immersed in the liquid, showed that the most rapid penetration occurred along the laminations. Therefore, to simulate practical conditions more closely, it was decided to expose the specimen to the liquid on one surface only.

The specimen (1 inch  $x\frac{1}{2}$  inch) is set in a thin layer of wax at the bottom of a small dish; three of the other surfaces are restricted with more wax, leaving only the inside concave surface exposed to the casting liquid (Fig. 3). The dish is stuck to a white card and lined up with the microscope in the same way as for the cordite granules, then placed in a constant temperature and humidity cabinet with the casting liquid in a separate vessel for 48 hours. On starting the test, the liquid is added carefully so as not to spill any on the top of the specimen.

In this case, two distinct zones are observed - an inner one, indicating the extent of penetration of the liquid, and an outer one, delineating the swollen boundary of the specimen. Both are measured with respect to the convex edge after 4 and 8 and 24 hours immersion and then daily for 10 days.

The colour of 2-nitrodiphenylamine is too intense to enable the swollen edge to be seen clearly, so it is not added to the casting liquid. However, as some contrast is necessary between the straw-coloured casting liquid and the clear specimen, the liquid is dyed a very pale green with an approved oil-soluble dye.

#### 3.3. The Dilatation of the Casting System

The dilatometer and associated apparatus, shown in Fig. 4, was designed to measure the change in volume which takes place after easting and during consolidation of a cast double base charge.

The casting vessel is a small glass cylinder with a sintered glass base and several lugs to support it in the dilatometer. The two sections of the dilatometer are joined by a "Quickfit" joint which, when coated with the appropriate grade of high vacuum grease (grade depending on the temperature), provides a watertight seal. The entry tube E and the top of the casting vessel are shaped so that the casting liquid will be delivered to the bottom of the dilatometer. The mercury serves two purposes, firstly, to displace the easting liquid into the capillary tube, and, secondly to wash the nitric ester from stopeock B and reduce the risk when the latter is closed.

The casting vessel is filled with a weighed amount of powder, a fine copper gauze restriction is placed on top, and the vessel is lowered carefully into the dilatometer. The apparatus is then assembled in the water bath, as shown, and given an hour at the required temperature to come to thermal equilibrium. With stopcock B open and screw clip F closed, vacuum is applied simultaneously

/to the

to the liquid reservoir and to the top of the capillary tube.

After evacuation for one hour the system is ready for casting, which is carried out as follows:

Screw clip C is closed and screw clip D opened to atmospheric pressure, then with the vacuum still applied to the capillary tube, F is slowly opened, admitting the casting liquid to the dilatometer. The rate of flow of liquid is regulated so that the level remains the same inside and outside the casting vessel. This is important as otherwise the liquid would flood into the top of the casting vessel before the casting is complete and create air bubbles which are difficult to remove.

When the dilatometer is nearly full, clip F is closed and the vacuum released. Mercury is run slowly in, until the liquid level in the capillary tube reaches a convenient height, when cock B is closed.

Fluctuations in the liquid level in the capillary are measured with a cathetometer, the top of the tube being taken as zero. The first few readings are taken at 5 minute intervals, then half hourly for 4 or 5 hours, and finally twice daily, until there is less than an 0.02% volume change between two readings.

#### 3.4. Densities of Chopped Cord and Cast Charges

The Jackson-Bianchi volumeter (Fig. 5) was used to measure the densities of casting powders, and of the propellants cast from them, at several temperatures, and from these results the coefficients of cubical expansion were calculated.

With this volumeter the volume of mercury displaced by the sample of either charge or powder is directly observed in a calibrated tube.

The apparatus consists of a glass volumeter with two calibrated capillaries, F and H, supported in a metal frame. Spindle S operates a reduction gear, to which the frame is attached, and enables the volumeter to be tilted slowly and evenly to a vertical position. A piece of plate glass C is held against the ground end of tube E by a hard rubber button on the end of a spring-loaded lever B.

Enough mercury is introduced into the volumeter to fill E and part of F when it is in a tilted position. For solid charges, the steel spring D must also be in position; for powders, spring D and the special wire gauze container which holds the sample of powder must be used. The apparatus is set up in an air thermostat at the required temperature and, with the weighed samples, is conditioned for 48 hours. The density measurements are carried out as follows.

With the volumeter in the horizontal position the glass plate C is removed, rubber bung A inserted in E, and both ends evacuated simultaneously. While still exhausted, the tube is tilted back until the mercury nearly reaches the top of E and entirely covers spring D (and wire gauze cage in the case of powder). Air is admitted through the dust filter M by means of the two-way tap N, the rubber bung is removed and the glass plate is replaced and clamped in position. The tube is further tilted until the mercury fills E completely, the remaining air escaping between the glass plate and the ground end of E, while the mercury is arrested. The position of the mercury surface is read on the graduated scale F.

The tube is then tilted back to the horizontal, C and D are removed, and the sample inserted in E. The steel spring is placed over the sample to prevent it rising through buoyancy. Evacuating and tilting are performed as before and a reading on scale H is obtained. The difference between the

readings is the volume of the sample.

It is important that both the sample and spring D are completely covered by mercury before admitting air to the exhausted tube.

Tilting should be slow and even, especially as the mercury begins to cover plate C. The glass plate C must be smooth and flat and the end of tube E ground perfectly flat, otherwise mercury will be lost.

At least three samples of each powder and charge were measured at 20°C., and 60°C.

#### 3.5. Packing and Packing Fractions

The highest nitrocellulose content of solventless cordite is normally 57% and it is doubtful whether this figure will be exceeded by existing methods of manufacture. Therefore, for the cast double base process using single base casting powder containing 88% - 90% of nitrocellulose, it will be necessary to achieve a packing fraction of at least 64% to match the highest nitrocellulose content of solventless cordite.

The chopped cord used for cast propellants is in the form of isodimetric cylinders which gave a maximum random packing of 67% and therefore will give a similar composition to that of solventless cordite.

The filling head Fig. 6.A, had been designed to give a high, uniform random packing for use with the 6 inch casting plant. The rate control plate has 7/32 inch diameter holes set at 1 inch centres, and the distributor plate has 7/64 inch diameter holes set at 3/16 inch centres. The rate control plate is designed to regulate the feed to the distributor plate and if the holes in it are smaller the feed will be slow for practical filling whilst if they are larger the rate of feed will be too fast and result in clogging.

The optimum ratio of diameter of the distributor plate holes to the dimensions of the powder (in this case 0.030 x 0.030 inch) is approximately 3 to 1 to prevent clogging and deliver the powder as a steady rain. The 20 inches from the lower plate to the casting beaker has been found to be the minimum fall required to give a packing fraction within 0.2% of the maximum.

In designing a 1 inch filling head for the long-tube casting apparatus, it was found that 7/64 inch holes in the distributor plate were too small and they had to be increased to 9/64 inch at 7/32 inch centres before clogging was eliminated.

Owing to variations in the sizes of the long glass tubes a slight taper was provided on the base of the 1 inch filling head.

### 3.6. Rate of Solution of Casting Powder in Casting Liquid

A direct investigation of the rate of solution of casting powder in casting liquid cannot be carried out, because it is not readily possible to determine quantitatively the small amounts of nitrocellulose involved. Also, as only partial solution takes place, there can be no direct correlation between viscosity and concentration because the lower molecular weight nitrocelluloses dissolve preferentially.

The really important factor from a practical point of view is not the increase in concentration but the increase in viscosity of the casting liquid due to solution of the nitrocellulose, as this is one of the limiting factors in casting long charges.

The increase

The increase in viscosity was measured as far as possible under practical casting conditions. A series of long-tube castings (Fig. 8) was carried out under various conditions of flow and temperature, samples of the advancing liquid were drawn off at several levels and their viscosities measured with an Ostwald U-tube viscometer. The nitrocellulose content is a maximum at the advancing liquid surface where there has been the longest contact between casting liquid and casting powder; therefore, these results will give the rate of increase in viscosity with time of contact under various operating conditions.

These results show only the viscosity changes at the advancing liquid surface. To investigate the effect on the viscosity as fresh liquid passes through the powder bed would require successive samples of liquid from the intermediate levels, which is not practicable with the long-tube assembly. Therefore, in order to be able to take successive samples of liquid at regular intervals, without interrupting the flow through the powder bed, the apparatus shown in Fig. 7 was set up.

This easting assembly is similar to that shown in Fig. 8, except that the column is only 90 cm. high, or equivalent to the first level of the long tube. The filling of the column, conditioning, evacuation and casting procedure are as described in Section 3.7. The casting liquid passes through the powder bed and out of the side arm into the graduated receiver, a sample (25 cc.) of the advancing liquid being taken after every 75 cc. have passed into the receiver. Therefore, samples are taken after every 100 cc. of liquid has passed through the powder bed, rather than at regular time intervals. The sample tube is thoroughly evacuated and clip A closed, then it is attached to the side arm below clip B. When the sample is required clip B is opened, followed by clip A. When sufficient liquid has been run into the tube, clip B is closed, the sampling tube removed and replaced with a clean evacuated one.

Samples are taken over a period of 40 to 50 minutes and the viscosities measured on the Ostwald U-tube viscometer.

These experiments were carried out at various temperatures but with no applied nitrogen pressure because the flow rate would have been too fast to permit sampling at regular intervals with such pressure.

#### 3.7. Long-Tube Castings

The apparatus shown in Fig. 8 was designed for the experimental casting of long colloidal charges to establish the factors affecting time and length of casting, and to determine the optimum operating conditions required for large-scale manufacture.

The column is a one inch I.D. glass tube 15 feet long, made by joining 3 five foot lengths of Pyrex industrial piping, to which 4 side arms are attached at 3 foot intervals for sampling the casting liquid. The tube is tested to withstand 80 to 100 p.s.i., which allows an ample margin for safety. The liquid inlet-tube at the base of the column is  $\frac{1}{4}$  inch I.D., and is connected to the liquid reservoir by  $\frac{3}{8}$  inch pressure tubing. The casting liquid reservoir is a copper vessel, capacity 1,500 cc. with  $\frac{3}{8}$  inch I.D. copper inlet and outlet tubes passing through a rubber bung, fitted into the top of the vessel and strapped into position. The reservoir is tested to 100 p.s.i. The nitrogen cylinder is fitted with the usual type of reducing valve and adjustable safety valve combined. The 'dry ice' trap consists of a large tube (8 x  $1\frac{1}{2}$  inches) fitted with a rubber bung. It sits on glass beads in the bottom of a vacuum-insulated vessel half filled with acctone and 'dry ice'. The inlet tube to the 'dry ice' trap (i.e., from the casting assembly) is 0.5 inch I.D., this larger size being necessary to prevent drops of water

/freezing

freezing on the tube and sealing off the vacuum pump. Connecting lines are of 3 inch pressure tubing.

The column is volume-calibrated in 100 cc. divisions with water, dried, then packed with powder through a distributor plate (Fig. 6) in 100 g. lots, the height reached in each case being marked on the tube with a paper label. These figures then give both the overall packing fraction and the individual packing fractions at any height.

The side arms are blocked with glasswool to prevent the powder falling into the sampling tubes. The powder is retained at the base and at the top by fine copper gauzes.

The column is surrounded by a 6 inch diameter cellulose acetate tube. It has a side arm at the base in which is fitted a glass coil for heating or cooling the forced draught induced by a small 5 inch fan. Small windows in the side of the tube allow access to the side arm sampling tubes. For low temperature castings on warm days the rather crude expedient of adding lumps of 'dry ice' to the side arm enabled the temperature to be maintained with only a 2°C. rise up the column. If the room temperature was fairly close to the required temperature it was possible to maintain the column at a temperature gradient of less than 1°C.

The casting procedure is as follows. The column of powder and the liquid in the reservoir are adjusted to the required temperatures and given two hours to come to equilibrium. With clip D open and clips A, B and C closed, vacuum is applied simultaneously to the column of powder and to the liquid reservoir and maintained at 2.0 mm. of mercury or below, for one hour. For casting at atmospheric pressure, clip D is closed and clip C opened to make the system ready. If applied nitrogen pressure is required the reducing and safety valves on the cylinder are set and clips C and D closed and B opened.

Casting is begun by opening clip A, simultaneously starting a stop watch. Times are taken as the liquid rises past the labels on the column and also when it runs into the sampling tubes. The time of casting varies, depending on the operating conditions, but it is usually about an hour. As the sampling of liquid interferes with the rate of flow, similar runs are always carried out without taking samples.

Castings have been carried out using a standard casting liquid with several grades of single base casting powder over a temperature range of 15° to 32°C. and a pressure range of atmospheric to 30 p.s.i. applied nitrogen pressure. Frequent duplicate runs showed that the reproducibility was very good. In each case the height cast was plotted against the time of casting.

#### 4. RESULTS

#### 4.1. Rate of Dilatation of Powder Granules

Dilatations were measured with single base casting powder (0.030 inch long x 0.030 inch diameter) in casting liquid consisting of 72.5% nitroglycerine and 27.5% triacetin at temperatures of 15°, 20° and 26°C.

#### The composition of Single Base Casting Powder No. 1 is:

Nitrocellulose (nitrated wood paper 12.2% N2)

Diethylhexyl phthalate

Lead stearate

2-Nitrodiphenylamine

Graphite (glaze)

88%

5%

2%

0.05%

The results

The results show that the dilatation is greater across the diameter of the granule than along its length and that for small increases in temperature there is a considerable increase in the rate of dilatation. As the temperature is raised the length of time during which the swollen edge can be measured accurately decreases, due to gradual disintegration when the percentage increase exceeds 200.

Fluctuations in the measurements due mainly to the irregularity of the surface of the granule make it essential to carry out at least 6 to 8 determinations on each axis of the granule to obtain reliable results.

In Table 1 are summarised the measurements of the percentage increases on both axes at the three temperatures for intervals up to 96 hours.

TABLE 1

Percentage Increase in Dimensions of Cordite Granules in Casting Liquid

Axis		Period of Immersion.							
Measured	Temperature.	4 hours	6 hours	24 hours	48 hours	72 hours	96 hours		
X	15°C.	1.0	1.7 2.4	10 14•5	20 35•5	36 55	53 77		
X	20°C.	3.3 4.0	5•3 6•5	25 30	50 73	79 97	114 132		
X	26°C.	13 21	17 29	65 106	97 <b>15</b> 8	134 195	_		

The plot of log (percentage increase in thickness) against log time gives a straight line relationship. Fig. 9 shows the rate of dilatation for the X axis and Y axis at 20°C., and Fig. 10 the effect of temperature on the rate of dilatation.

It will require a 14.6% increase in the dimensions of 0.030 inch x 0.030 inch powder to increase the volume by 50%, or sufficient to fill the voids completely.

# 4.2. Dilatation of, and Penetration into Cellulose Acetate, by Casting Liquid.

Several samples of convolute wound cellulose acetate containers, \$\frac{1}{8}\$ inch thick, produced from film-quality foil, were measured for dilatation and penetration in casting liquid consisting of 72.5% nitroglycerine and 27.5% triacetin. The composition of the foil was as follows:

#### B.X. Plastics Film Quality Foil:

Cellulose acetate	80.0%
Triphenyl phosphate	20.0%
Acetyl content (as CH3. COOH)	57.5%
Viscosity of 1% solution relative to acctone at 20°C.	4.27

The average percentage rates of increase in dimensions and penetration from these experiments are given in Table 2.

Percentage Increase of Wall Thickness and Penetration of
Casting Liquid into Cellulose Acetate.

Time in Hours	4	7	24	48	72	96	168	192,
Dilatation	5%	8.1%	15.2%	20.6%	29.4%	39%	48.5%	50%
Penetration	3%	3.4%	7.2%	9.5%	12.6%	15%	18.6%	19.1%

The log (percentage increase) plotted against log time gives a straight line relationship in each case and these are shown in Fig. 11.

These results are used as a standard for comparison with other samples of convolute-wound containers using different bonding solutions. Increasing the plasticiser content increased both the dilatation and penetration and the use of a crosslinking agent slightly increased dilatation and in addition caused irregular penetration giving the sample a corrugated appearance.

The addition of 10% hexamethylene di-isocyanate to the bonding solvent as a crosslinking agent resulted in the container dilating 67.6% after 150 hours immersion.

#### 4.3. Volume Change During Consolidation

Differences in the calculated and measured densities of charges cast in the 6 inch pilot plant have indicated that the casting process is accompanied by a real loss in volume. Typical figures obtained for charges cast with single and double base casting powders are shown in Table 3.

TABLE 3

Calculated Volume Decrease on Curing - 6 inch Plant.

Density of Components, g/cc.		Weight Ratio		Density of Ca		
Powder	Liquid	Solid.	Liguid	Calculated from Columns 1 to 4	Measured	Percentage Loss in Volume
Single base - 1.552	1.450	66.5	33.5	1. 518	1.543	1.81%
Double base - 1.603	1.370	65.6	34.4	1. 521	1.531	0.66%

A series of small charges were cast inside the dilatometer. Casting and curing were carried out at the same temperature to avoid the influence of thermal expansion. Charges were cast using single base and double base powders, with their respective casting liquids, at 45°C. and 60°C. The composition of these casting powders and liquids is given in Table 4.

TABLE 4
Casting Powder and Liquid Compositions

	Single Base	Double Base	Casting Liquid		
Component.	Powder, F.452/40.	Powder, F.428/183	Used with F.452/40.	Used with F.428/183	
Nitrocellulose (12.2% N)	88%	74%		la.	
Nitroglycerine		20%	72%	64%	
Triacetin			27%	35%	
Diethyl hexyl phthalate	5%				
.Diethyl phthalate		5%	in.		
2-Nitrodiphenylamine	2%				
Lead stearate	5%				
Carbamite		1%	1%	1%	
Carbon black		0.05%			
Graphite glaze	0.05%				

A further series was cast at the normal casting temperature of 20°C. and maintained at this temperature to determine the loss in volume occurring without appreciable curing taking place. Duplicate castings showed good agreement and the final results are recorded in Table 5.

/Table 5.

# TABLE 5 Volume Decrease on Curing.

Temperature	Time	Single Base Powder	Double Base Powder
20°C.	220 hours	0.69%	0.47%
45°C.	120 "	0.52%	0.25%
60°C.	100 "	0.42%	0.23%

The curves in Fig. 12 show the rate of decrease in volume during curing while Fig. 13 shows the change taking place after casting without the influence of curing.

#### 4.4. Volume Coefficients of Powders and Charges

The densities of single and double base casting powders and the propellants cast from them were determined and from these results the coefficients of cubical expansion were calculated and are shown in Table 6. The temperature range 20° to 60°C. was selected as being normal between casting and curing. A check at 45°C. showed the coefficient to be constant between 20°C. and 60°C.

TABLE 6

Coefficients of Cubical Expansion of Powders and Charges

Material	Coefficient of Cubical Expansion per °C,
(a) Single base casting powder  (b) Double base casting powder	32 35•5
(c) Cast propellant containing (a) (d) Cast propellant containing (b)	43 40. 5

Thus on casting at 20°C. and curing at 60°C. the thermal expansion for propellant cast with single base powder will be 1.72% and for propellant cast with double base powder 1.62%.

In both cases this is partly offset by the real loss in volume occurring during casting and curing, and therefore the overall expansion will be less. (Approximately 1.1% in each case, based on dilatometric measurements. Measurement of 6 inch plant charges cast with single base powder gave zero and charges cast with double base powder approximately 1%).

#### 4.5. Rate of Solution of Nitrocellulose in Casting Liquid

Using a single base casting powder (F.452/40) and casting liquid of composition 72% nitroglycerine, 27% triacetin and 1% 2-nitrodiphenylamine, a series of long tubes were cast at 15°, 20° and 26°C. as described in Section 3.7. Samples of the advancing liquid were taken and viscosities measured. The results are set out in Table 7 which gives the time of contact and the viscosity of the liquid (in centistokes) for castings at each temperature.

TABLE 7

Effect of Temperature on the Viscosity of Casting Liquid

	Viscosity of Liquid at: #					
Time of Contact	15°C.	20°C.	26°C.			
2 minutes	20.6 cs.	21.5 cs.	23.0 cs.			
5 "	21.7 "	23.6 "	28.3 "			
10 "	23.5 "	27.7 "	41.4 "			
15 "	24.8 "	32.5 "	62.8 "			
25 "	27.4 "	45.0 "	130 "			
32.5 "	29.3 "	57.5 "	176.5 "			
40 "	31.3 "	73.8 "	-			
60 "	-	-	gels.			
90 "	-	gels.				
120 "	gels.		A			

<sup>\*</sup> Viscosity of original casting liquid = 19.7 cS.

The times quoted in Table 7 at which the casting gels, represent the ultimate stage, when the flow has ceased, but the 'slow up' to this stage is considerable, and can be gauged from the curves in Fig. 20.

The viscosity has been plotted against the time of contact for the three temperatures and the resulting curves are shown in Fig. 14.

It can be seen that successive sampling of the long tube castings would not be practicable, so a series of short tube castings were carried out as described in Section 3.6. Samples were taken in each case after 100 cc. of fresh liquid had passed through the powder bed, and though the contact times are not the same the viscosities of the samples provide some interesting data, which are set out in Table 8.

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TABLE 8

ratio other

# Change in Viscosity of Fresh Casting Liquid Passing through the same Powder Bed

180 30	Casting	at 15%.	Casting	at 20°C.	11000	at 260c.
Liquid passed through Powder Bed.	Contact Time,	Viscosity,	Contact Time,	Viscosity,	Contact Time,	Viscosity,
c.c.	min.	cS.	min.	oS. Va	min. n	oS.
100	- 6	21.5	5'^'	. Tr. 22001 to	ri <mark>t</mark> ion	26.8
200	7	25.6	7	27.2	61/2	36.2
300	8	25.7	7	29.5	niologia bas nomio	40 3 F
400	9	25.0	8 7	29.23	l toegivo	38.9
500	10	24.9	-ng 15n	28.7	okun <b>01</b> 010 neeim at	40.4
600	10	24.9	9.5.		inneyq off	42:7
700	10	25.1	10	28.9 W	toofing file	7 CH 147.7
800	10	25.4	11		of 25 ment	63.60
Jahania 900 Subarry		25.1	12	- 10 J	oria govin	enould not
Overall time of contact.	82	- - #12-fe	78 ********			wnil c'her The r

In the case of the eastings run at 15° and 20°C., the casting liquid was still flowing steadily through the powder bed after 82 and 78 minutes, respectively, but with the one run at 26°C. the flow of liquid was slowing up considerably after 88 minutes. It will be noted that the viscosities are much less than those at a corresponding stage for the advancing liquid in the long tube castings.

As it was not possible to determine small quantities of nitrocellulose in casting liquid, there was no way of finding the actual percentage dissolved in the liquid and carried upwards during casting. However, a series of control solutions of single base casting powder in easting liquid were made up at increasing concentrations, and the viscosities measured.

As the powder is very slowly soluble in casting liquid a standard solution was made up in acetene. Requisite aliquots were added to portions of casting liquid and these evacuated until all the acetone had been removed. A fine stream of air and gentle warming (35°C.) were used to assist removal of the volatile solvent. The highest concentration made up was 0.2%, based on the nitrocellulose content of the casting powder. A plot of viscosity against concentration is shown in Fig. 15.

/It was

mont lightly

It was considered doubtful whether this graph could be used to predict the percentage of nitrocellulose dissolved during casting because of the possibility of preferential extraction of the lower molecular weight nitrocelluloses.

Should the sharp rise in the viscosity represent an appreciable increase in the nitrocellulose content it would result in the top of the charge having a higher concentration than the base; in fact the analysis of sections of numerous castings showed that the maximum variation in composition along the length of the charge was 1%.

#### 4.6. Packing Fraction of Powders

Random packing by means of a filling head incorporating a distributor plate (Section 3.5.) gives a higher uniform packing fraction than is obtained by other methods, employing mechanical shakers or vibrators. Vibrators have the very undesirable effect of creating nodes of different density which yield castings of uneven composition.

It has also been found that the height of fall of the powder from the distributor plate has an influence on the packing fraction. The latter increases with the former and 20 inches is considered to be the minimum height the powder should fall to give a satisfactory packing fraction (Fig. 16).

In packing glass tubes 15 feet long, 1 inch diameter, the variation in packing fraction at any individual level from the overall average was less than ± 0.25%. The overall average varied less than ± 0.5% from tube to tube.

The wall effect with the 1 inch tubes was considerable and the average packing fraction obtained was only 65.6%, compared with an average of 67.3% for the 6 inch pilot plant.

For a given shape of powder variations in the size, within practical limits, should not alter the packing fraction, providing each size is uniform and the wall effect negligible.

The more efficiently a casting powder is graphited the closer its packing fraction will approach the limiting value.

A number of 15 foot columns were packed with 99.5 g. lots of black-coloured powder interspaced with 0.5 g. layers of red-coloured powder. The positions of these layers were carefully marked on the glass tubes and observed during subsequent filling. The results show that the packed beds were not compressed by the weight of the column of powder.

#### 4.7. Effect of Pressure on Casting

In order to determine the flow of liquid through the powder bed at various pressures without the influence of dilatation, or solution of the casting powder, dummy castings were carried out using glycerine and water of the same viscosity as casting lquid. Procedure for these dummy castings was the same as that described in Section 3.7. and the results are shown in Fig. 17. When casting commences the surface of the liquid in the reservoir is level with the bottom of the bed of powder so that as casting proceeds the decreasing head of liquid is tending to slow down the rate of casting. Correcting for this head of liquid and plotting the corrected height cast against time on log-log paper the exponential functions shown in Fig. 18 are obtained for the three pressures.

The heights cast at various times are of the same order as calculated from

/a modified

a modified Kozeny equation. These heights also agree with the 6 inch plant castings, in which dilatation and rate of solution of the casting powder has not started to influence the rate of flow.

A series of actual long tube eastings were run at 20°C. at three pressures and the resulting curves are shown in Fig. 19. The slopes of the curves are very similar to those in Fig. 17, but when corrected for pressure loss and transposed to log-log paper they were only exponential functions for times up to 20 minutes. It is obvious that from this time onwards the rate of solution or dilatation is beginning to have an influence on the rate of easting.

Castings at the three pressures were also run at temperatures of 15° and 26°C, and all the results are set out immTable 9.

# TABLE 9 Effect of Pressure on Height Cast.

Oc. F

Height cast in cm.

	15°C.			20°c.			26°C.		
Casting Time,	Pressure			Pressure			Pressure of		
, min.	Atmos- pheric	15 p. s. i.	30 p.s.i.	Atmos- pheric.	15 p.s.i.	.30 p.s.i.	Atmos- pheric	15 p.s.i.	30 p.s.i.
5	-78	-115	145	92	134	172	1.00	150	187
10	112	164	204	122	179	230	129	196	245
15	127	193	238	143	211	272	149	225	280
20	142	216	267	159	235	304	160	240	307
25	155	234	291	172	256	331	169	260 .	- 324
30	165	248	311	183	272	355	175	270	337
40	180	271	347	199	297	395	185	288	358
50	194	291	384	211	316	422	_	雅二( *	-
60	206	306	407	220	332	44.5	-	- (c.)	-

As the curves of height cast against time are partially exponential there should be a relationship between height cast and pressure for time up to 20 minutes. In Table 9 the heights at atmospheric pressure were taken as unity and the heights at 15 and 30 p.s.i. applied pressure were calculated as multiples of these heights: the results are set out in Table 10.

/Table 10

TABLE 10

Relationship between Height cast and Applied Casting Pressure.

	15 <sup>0</sup> C. Pressure			20°C. Pressure			26°C. Pressure		
Casting Time,									
min.	Atmos- pheric	15 p.s.i.	30 p.s.i.	Atmos- pheric	15 p.s.i.	30 p.s.i.	Atmos- pheric	15 p. s. i.	30 p.s.i.
5	1	1.50	1.86	1	1.45	1.87	1	1.50	1.87
10	1	1.49	1.84	1	1.46	1.88	1	1.51	1.90
15	1	1.51	1.87	1	1.47	1.90	1	1. 51	1.88
20	1	1.52	1.88	1	1.48	1.91	1	1.50	1.92
25	1	1.51	1.88	1	1.49	1.92	1	1.52	1.91
30	1	1.51	1.89	1	1.48	1.93	.1	1.54	1.93
40	1	1.50	1.92	1	1.48	1.95	1	1.56	1.94
50	1	1.50	1.97	1	1.50	2.0	-	-	-
60	1	1.48	1.97	1	1.51	2.02	-	-	-

These results will provide a convenient means of predicting approximately to what height a given powder will east, providing it has been east at another pressure.

#### 4.8. Effect of Temperature on Casting

Raising the temperature of the casting system has a threefold effect:

- (a) The fluidity of the casting liquid is increased.
- (b) The rate of solution of nitrocellulose in casting liquid is increased.
- (c) The rate of dilatation of the powder is increased.

For charges up to 7 feet in length the fluidity is the dominant factor and there is a small saving in time by casting at a higher temperature. But in the region of 9 to 10 feet the effect of fluidity is cancelled by the increase in viscosity of the casting liquid due to the solution of nitrocellulose.

The times taken to cast C.P.3. charges (Single Base Casting Powder No. 1) to various heights are given in Table 11.

/Table 11

TABLE 11

Effect of Temperature on Height Cast

The state of the s		Filler	The state of the s	THE PARTY			
Height Cast,	Time Taken in Minutes.						
feet	Cast at 15°C.	Cast at 20°C.	Cast at 26°C.	Cast at 32°C.			
7	11½	8 <del>1</del>	7	61/2			
8	16	11½	10	104			
9	21 1/2	15½	14:	18½			
10	28 <del>1</del>	2014	20	gelled.			
11	36 <del>3</del>	25 <del>3</del>	29	and the state of			
12	47	32	47	4- 0- i-			
13	57 <del>1</del>	40 <del>1</del>	gelled.				
14	73	.50		Visc. 10 to 10			
15	97	65	The state of the s	ioniko labid k Liebi uska			

From Table 11 it can be seen that there is no advantage in casting charges in excess of 10 feet at temperatures above 20°C. Though it is possible to cast charges up to 15 feet at both 15°C. and 20°C. the casting at 15°C. is very slow due to the reduced fluidity of the casting liquid. Therefore 20°C. may be regarded as the best temperature at which to carry out all long castings.

The curves of the castings at the above four temperatures are shown in Fig. 20.

#### 4.9. The Effect of the Properties of a Powder on its Castability

The shape and size-distribution of the powder granules affects the packing fraction and therefore must also influence the height to which a charge can be cast.

The effect of a difference of 1.8% in the packing fraction on the height cast can be seen in Fig. 21 in which chopped cord (0.030 inch x 0.030 inch isodimetric cylinders), packed to different densities, was cast under similar conditions.

Powders of the same shape, but of different sizes, although they pack to the same density, will not cast to the same height. Considering two isodimetric cylindrical powders of size '0.030' and '0.035', when packed the larger size powder will have a lesser number of interstices than the small sized powder but they will be of greater volume. The smaller interstices will offer greater resistance to flow, and hence, a shorter height will be cast in a given time.

/By packing

By packing these powders to the same density and using glycerine and water as a casting liquid, thus eliminating solution and dilatation effects, the influence of this factor can be gauged. Curves A and B in Fig. 22 illustrate this influence.

There will also be a 35% reduction in surface area with the larger sized powder, which will considerably reduce the rate of solution of nitrocellulose in the casting liquid and materially assist the casting. Curves C and D in Fig. 22 represent runs cast with nitroglycerine casting liquid and these two different sized powders. The divergence between A and C and between B and D represents the effect of dilatation and rate of solution.

#### 5. DISCUSSION

Whilst the method of carrying out the dilatation measurements is not directly comparable with practical casting conditions, it provides an indication of the relative values of changes in powder composition, casting liquid and temperature. At temperatures of 15°C. and 20°C. the dilatation of Single Base Casting Powder No. 1 will have a negligible effect on the rate of casting because in the first hour the volume increases only 3%. However, as the temperature is raised to 26°C. the increase for the same period is 14%, or the third that required completely to fill the voids, and this will definitely influence the casting rate. Further measurements at higher temperatures should establish whether dilatation becomes the dominant factor or whether it continues to be a secondary one in the gelling of long castings. In the case of double base powders dilatation is a major factor; it has been shown that voids may be filled within an hour.

The ideal convolute-wound container, in which the charge is cast and which subsequently forms the restriction to prevent the outer cylindrical surface from burning, should bond perfectly to the charge without subsequent migration of the solvent, plasticiser or nitric ester. However, to obtain a good bond of container to charge some migration of one or more components must occur between the two during curing of the charge. The dilatation and penetration of the restrictive container by casting liquid will not affect the rate of casting but will influence bond of the charge to the container. The solution of the container material in the nitric ester will not affect the rate of casting appreciably, but may influence the quality of the bond. A study of the comparative rates of solution of cellulose acetate in the nitric esters may explain why nitroglycerine gives a good bond but diethylene glycol dinitrate a poor one with cellulose acetate restrictions.

The real losses in volume on easting and curing as measured by the casting of small charges in the dilatometer, are less than those obtained by measurement and weighing of a number of 6 inch diameter charges before and after curing, but follow the same pattern. The discrepancy may be due to the small charges not being restricted along their length and to the greater excess of liquid in the dilatometer. When the components are east and cured at a high temperature they contract less than if the operation is carried out at a lower temperature and also the more highly gelatinised 20% double base powder contracts less than the single base powder. To investigate the problem further it will be necessary to alter the design of the dilatometer so that a small charge can be east at 20°C. cured at 60°C., and cooled again, the volumes being measured before curing and after cooling. To do this will require the insertion of a calibrated bulb in the middle of the capillary of sufficient capacity to account for the thermal expansion of the liquid in the system.

The packing fraction governs the solid: liquid ratio and therefore controls the composition of the charge. For this reason it is essential that the method

of filling

of filling the restrictive containers with easting powder should give a high, uniform and reproducible packing density. A very high packing fraction, even if obtainable, is not practical because of the reduction in free space and the subsequent slowing down of the rate of easting. Nor is a low packing fraction desirable because of the resulting high percentage of liquid in the easting. Increasing the size of the powder without altering the shape will increase the rate of easting but will not altogether solve the problem of producing long eastings in short times, because of the disproportionate increase in the curing time.

The sharp rise in the viscosity of the advancing liquid is almost certainly the limiting factor in the easting of long (single base powder) charges. The nitrocellulose in the casting powder has a wide range of molecular weights of varying solubilities in the casting liquid. To what extent the low molecular weight nitrocelluloses are preferentially dissolved cannot be ascertained until it is possible to determine quantitatively the small amounts of nitrocellulose in the casting liquid. The results of the experiments in which fresh liquid was continually passed through the same powder bed show that the viscosities of successive samples do not vary greatly, providing the contact times are the same. This means that the concentration of nitrocellulose in these samples is either uniform or is decreasing with the solution of higher molecular weight nitrocelluloses of correspondingly higher viscosity. This work will have to be extended, and the study of rates of solution of nitrocellulose in other nitric esters included, before a clear picture can be obtained.

Considering the results of castings carried out at different temperatures, there is little doubt that 20°C. is the best practical temperature at which to cast charges up to 12 feet. The slight gain in time obtained by casting shorter charges at a higher ambient temperature is not worth the risk of the charge gelling should casting be interrupted for a few minutes by a power failure or other causes. The acceptance of 20°C. as the optimum casting temperature means that provision will have to be made for heating and cooling the casting assembly, or conditioning the casting house.

The result of experiments involving only changes in pressure show that increasing pressure directly increases the length to which a charge can be cast. Therefore, the limiting positive pressure at which a charge can be cast will be dependent mainly on the feasibility of handling nitric esters under pressure. Until more information is available as to the safety of such handling it is considered that a positive operating pressure of 30 to 35 p.s.i. should not be exceeded.

The experimental casting system is evacuated and maintained for 1 to 2 hours at 2.0 mm. Hg or less to remove moisture from the surface of the granules and to dry and de-aerate the casting liquid. This period of evacuation is very important, because traces of moisture may cause swelling without solution, resulting in a 'boiled rice' effect and poor cohesion. It has been suggested that during the period of evacuation some surface conditioning of the granules occurs which improves the quality of the casting. Further work on this possible mechanism is required.

#### 6. SUMMARY OF RESULTS

6.1. The dilatation of single base easting powder granules is greater along the Y axis (diameter) than along the X axis (length) and the rate of dilatation increases rapidly with a rise in temperature. Dilatation is an exponential function of time up to 100 hours immersion.

16.2.

- 6.2. Standard cellulose acetate convolute-wound containers dilate 50% and are penetrated 20% after 200 hours contact with casting liquid. Both dilatation and penetration are exponential functions of time for this period.
- 6.3. There is a real loss in volume on casting and curing, which is greater for single base than for double base powders. This loss in volume, as measured after 200 hours, decreases as the curing temperature is raised.
- 6.4. Based on dilatometric experiments, the overall thermal expansion of charges cast at 20°C. and cured at 60°C., taking into account the irreversible loss in volume, is approximately 1.1%. Practical charge measurements give a lower figure.
- 6.5. For high, uniform packing, when a filling is done through a distributor plate having holes of a fixed size and spacing (related to the dimensions of the powder) it is essential that the casting powder falls at least 20 inches.
- 6.6. As the casting temperature is raised the viscosity of the casting liquid rises steeply due to the increased rate of solution of nitrocellulose in the liquid and ultimately causes the casting to gel. With C.P.3 composition gelation occurs after 129, 90, 60 and 30 minutes at temperatures of 15°, 20°, 26° and 32°C., respectively.
- 6.7. Applied pressure directly influences the height to which a charge can be cast and for short times of casting it is possible to predict approximately, other things being equal, what increase in height will be obtained with a known increase in pressure.
- 6.8. The maximum useful casting time falls rapidly as the temperature rises from 15°C. to 32°C., but taking fluidity, rate of solution of nitrocellulose and easting time into account, 20°C. is the best practical temperature for casting charges up to 15 feet long.
- 6.9. Increasing the size of powder of a given composition whilst retaining the same shape will increase the height of casting, but this is not altogether advantageous because the curing time is considerably increased.

#### 7. ACKNOWLEDGEMENTS

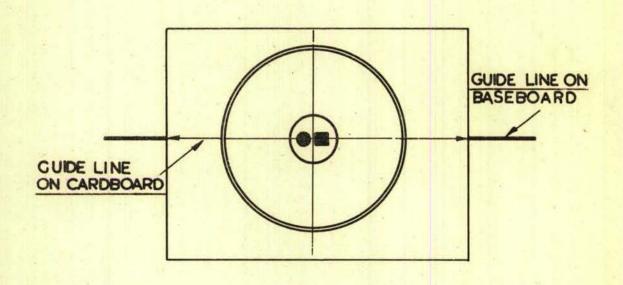
This work was initiated and supervised by Mr. W.N. Hewson to whom the author is indebted for many helpful discussions and useful suggestions. The author also thanks Mr. A.J. Thomas for his advice with the work on dilatation and Mr. R.J. Walley for data supplied relating to the 6 inch experimental casting plant.

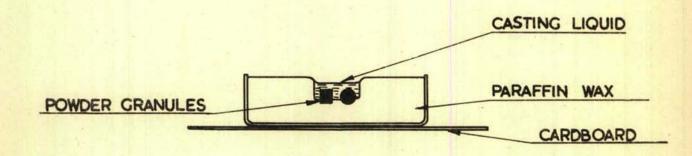
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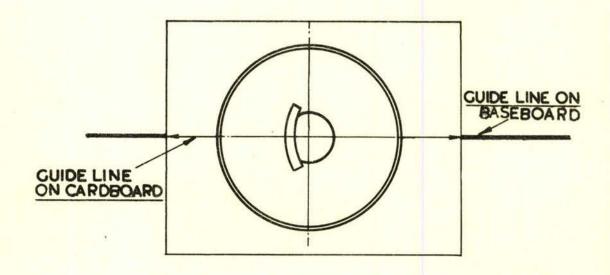
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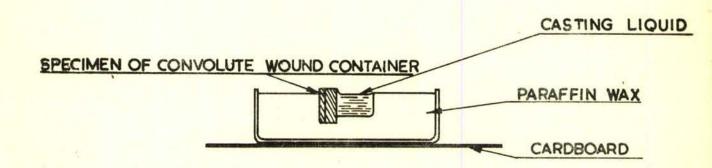




DILATATION OF POWDER GRANULES

FIG. I.





# DILATATION OF CONVOLUTE WOUND CONTAINER FIG 3

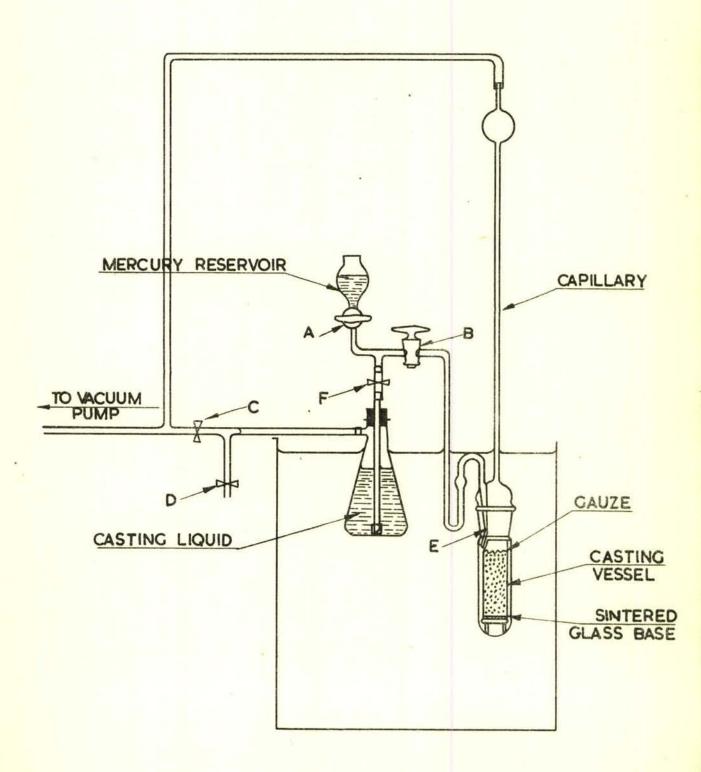


FIG. 4. DILATATION OF THE CASTING SYSTEM

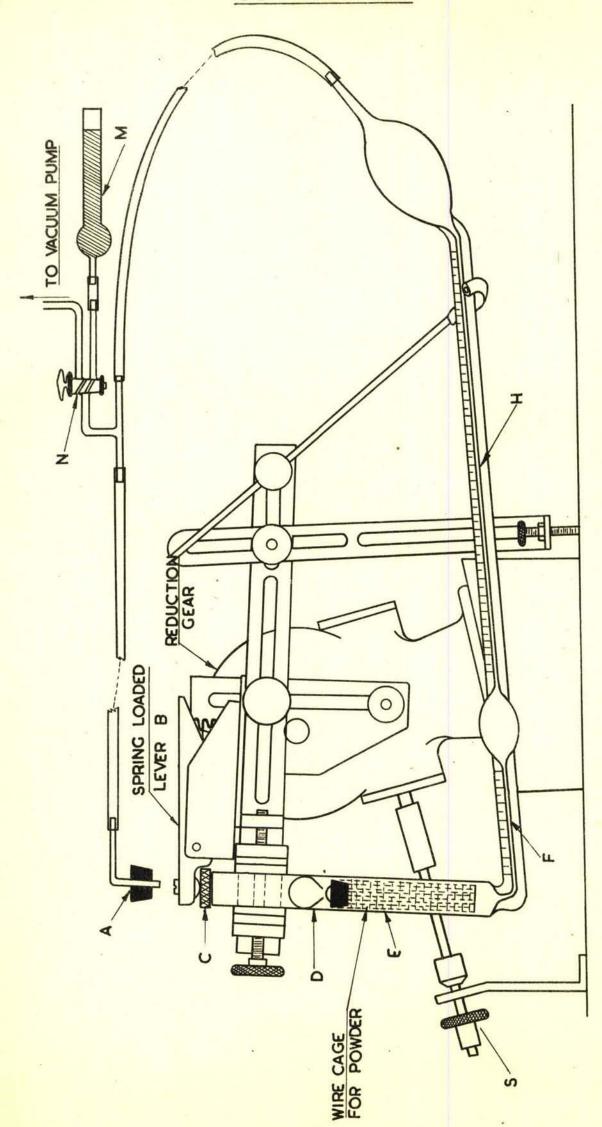


FIG. 5. JACKSON - BIANCHI MERCURY DISPLACEMENT VOLUMETER

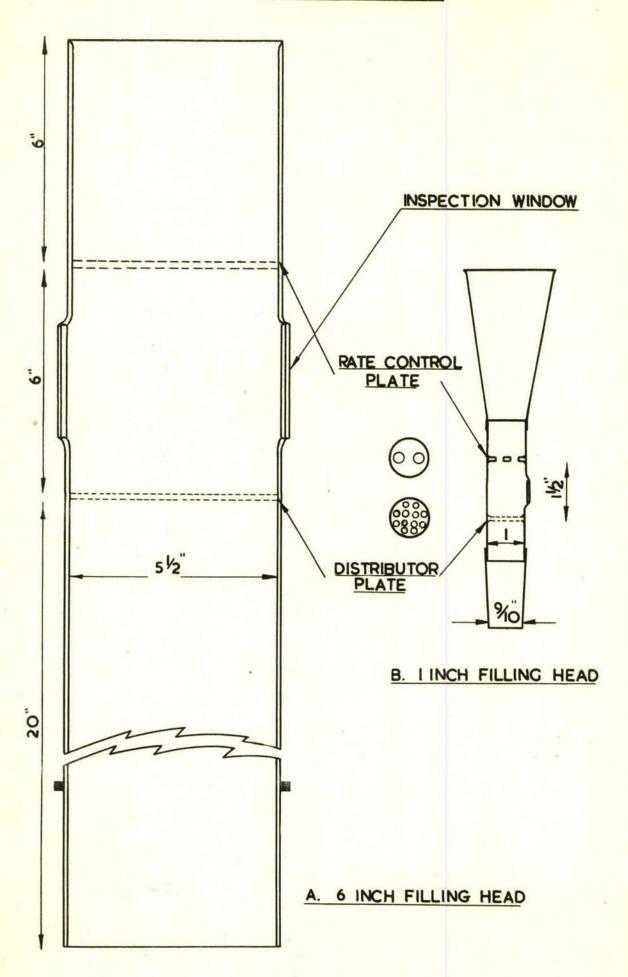
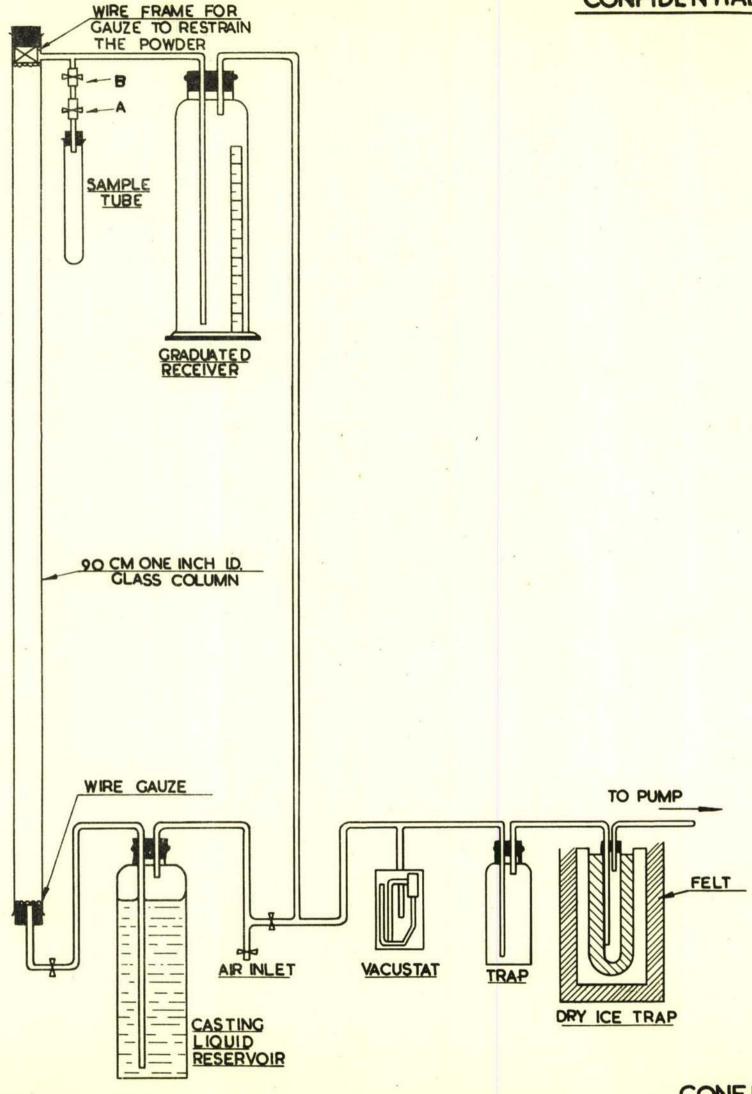


FIG.6. FILLING HEADS



APPARATUS FOR SUCCESSIVE SAMPLING OF CASTING LIQUID. FIG.7.

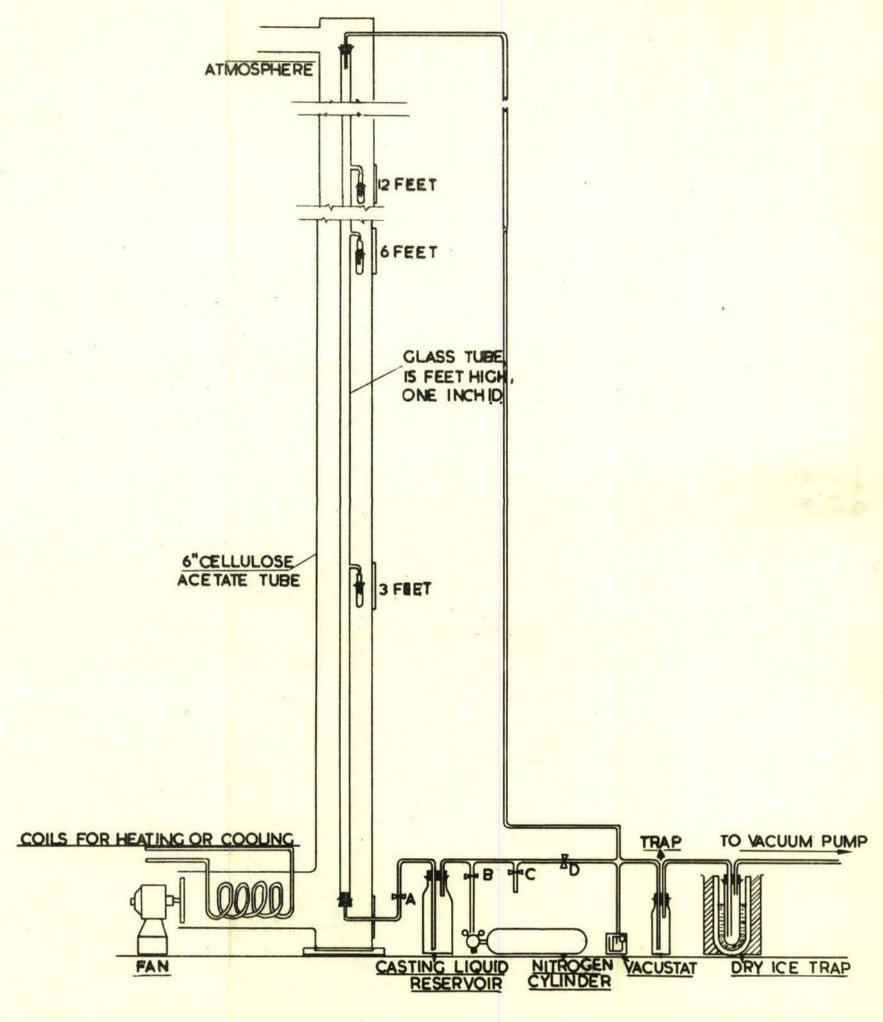
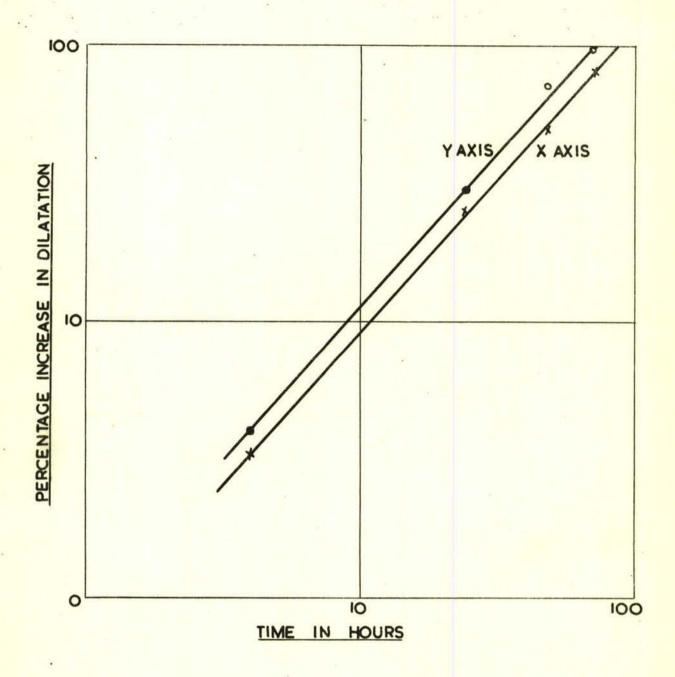
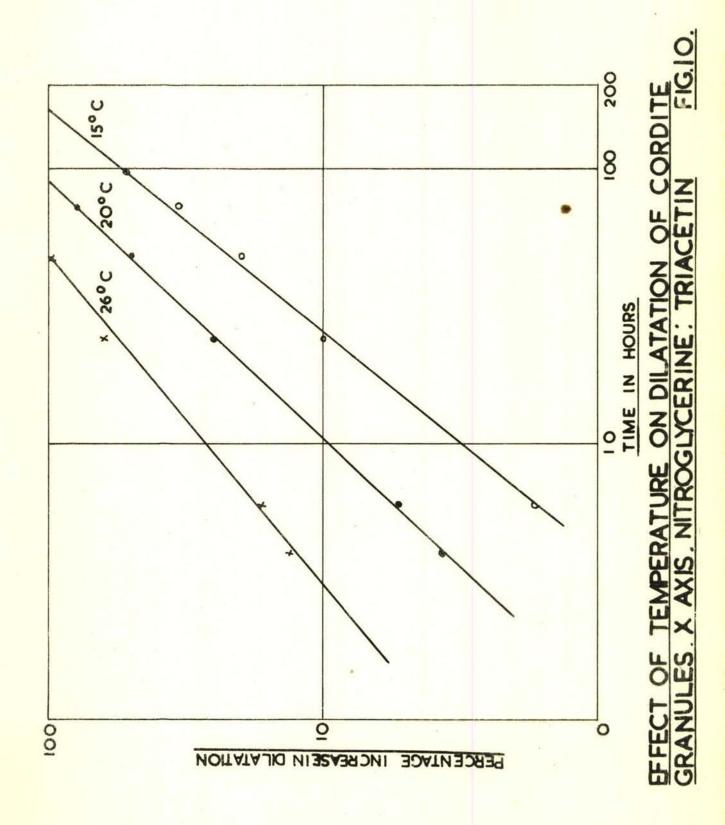


FIG. 8. APPARATUS FOR EXPERIMENTAL CASTING OF LONG TUBES

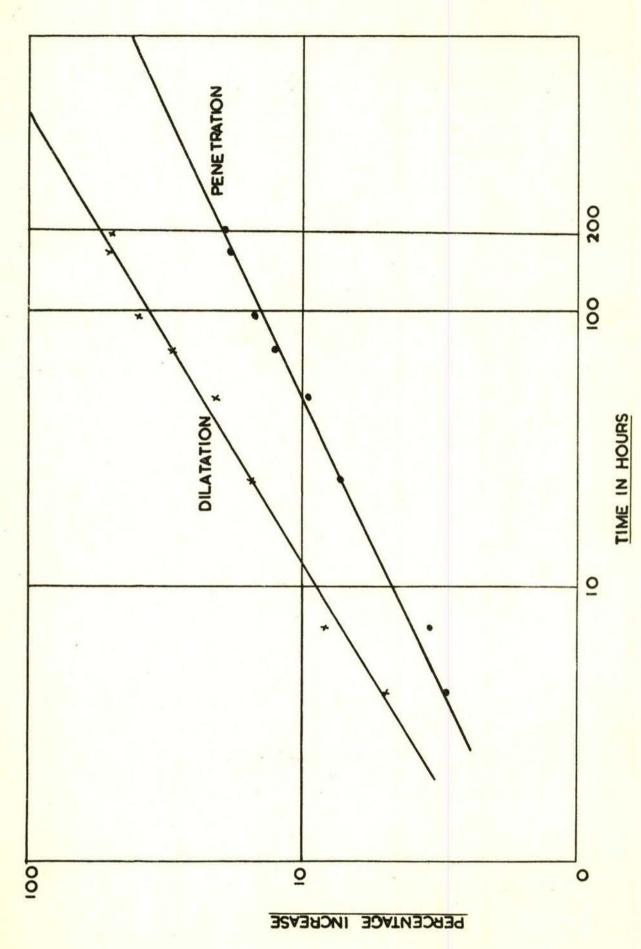


## DILATATION OF CORDITE GRANULE IN CASTING LIQUID 0.030 INCH X 0.030 INCH AT 20°C

FIG. 9



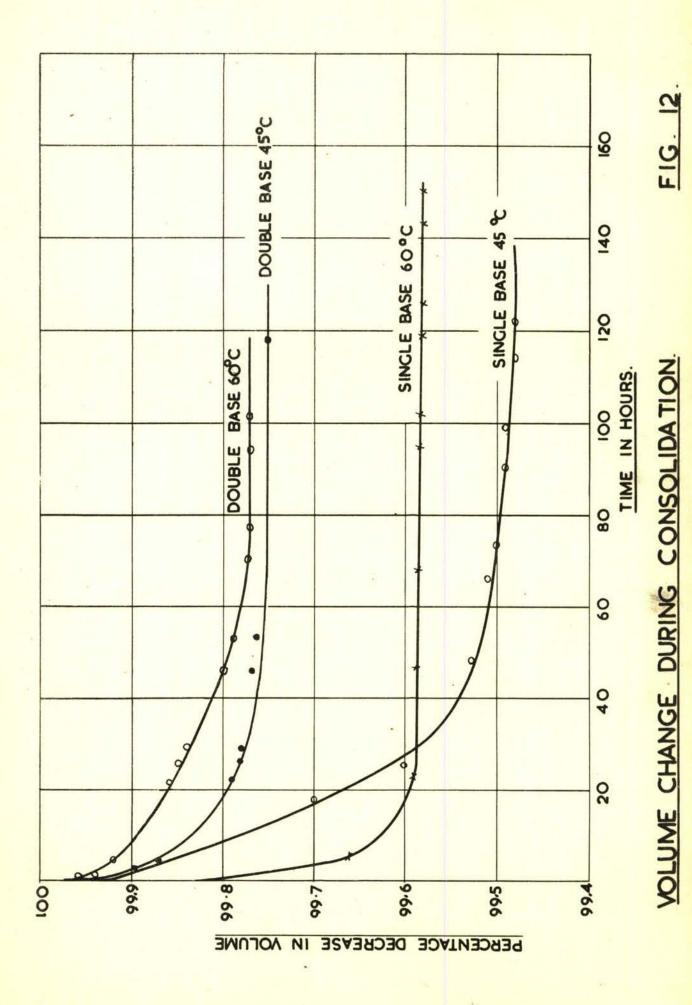
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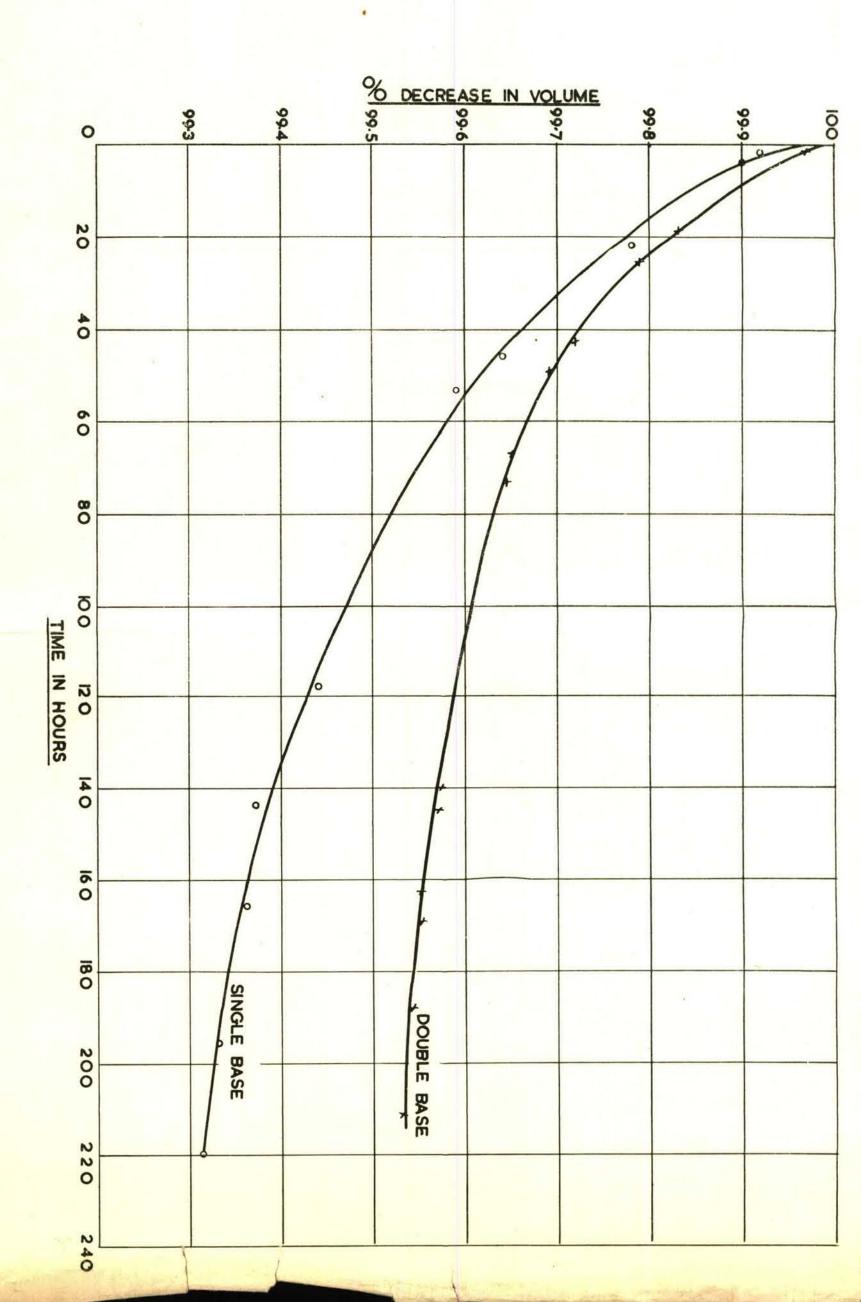
DILATATION AND PENETRATION OF CELLULOSE ACETATE AT 20° C

FIG. II.

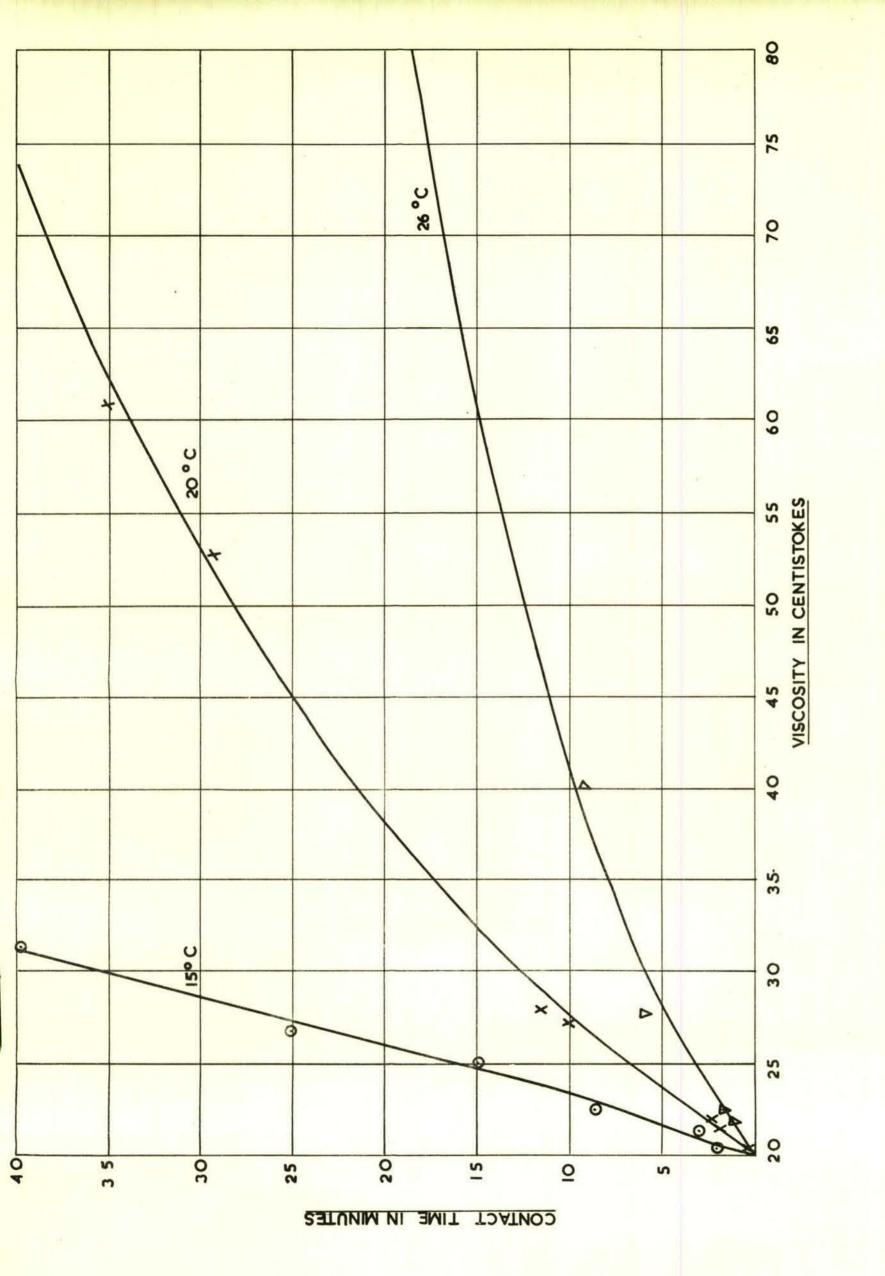
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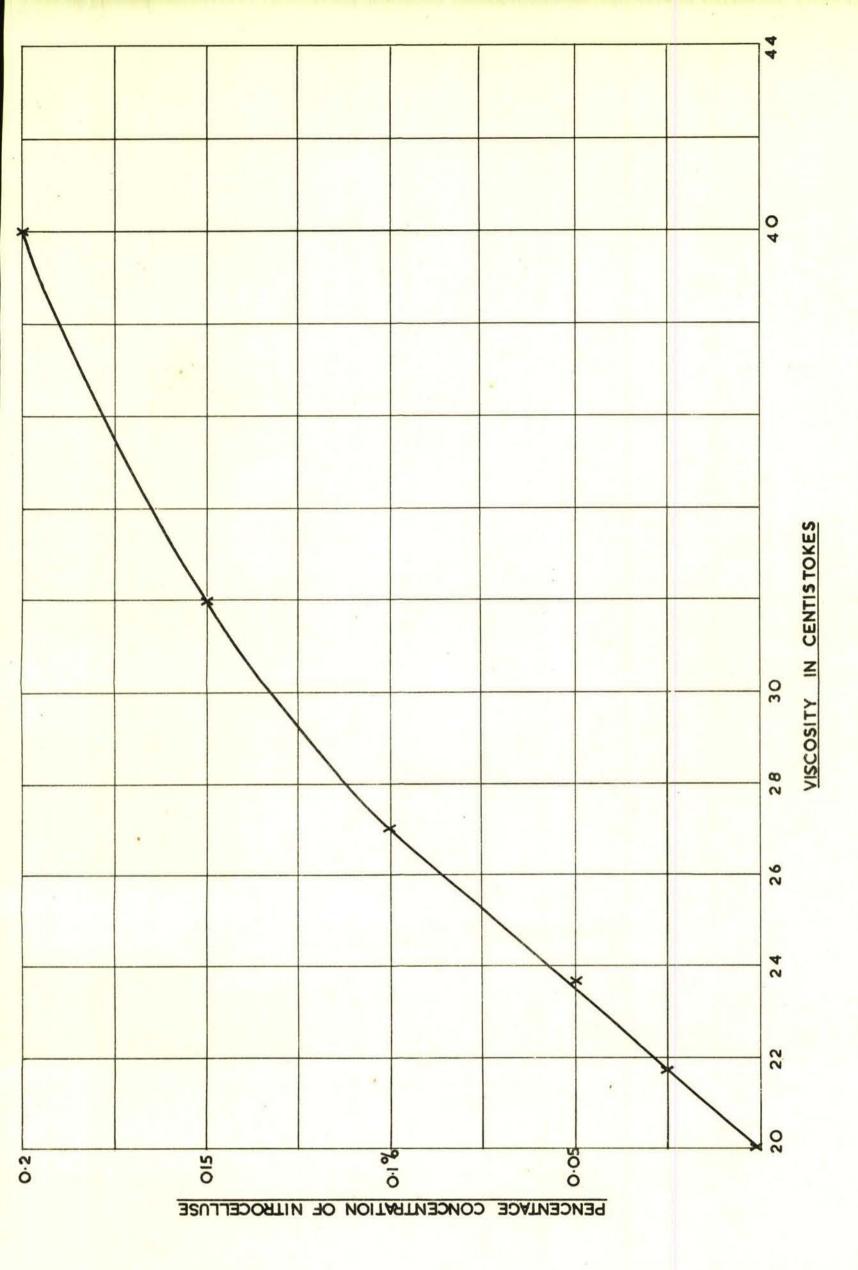


VOLUME CHANGE ON CASTING, 20° C - NO APPRECIABLE CURING, FIG.13



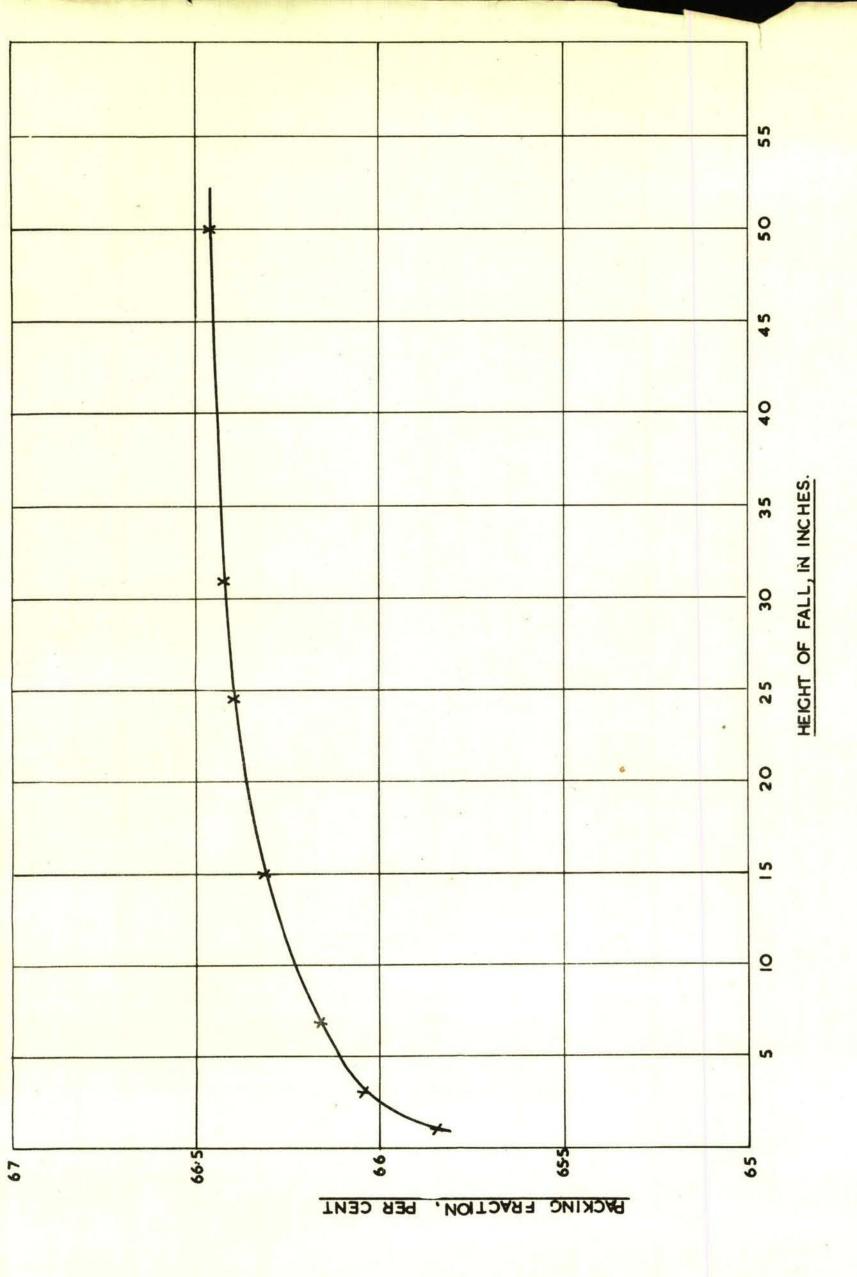
EFFECT OF TEMPERATURE ON VISCOSITY OF CASTING LIQUID.

FIG . 14.



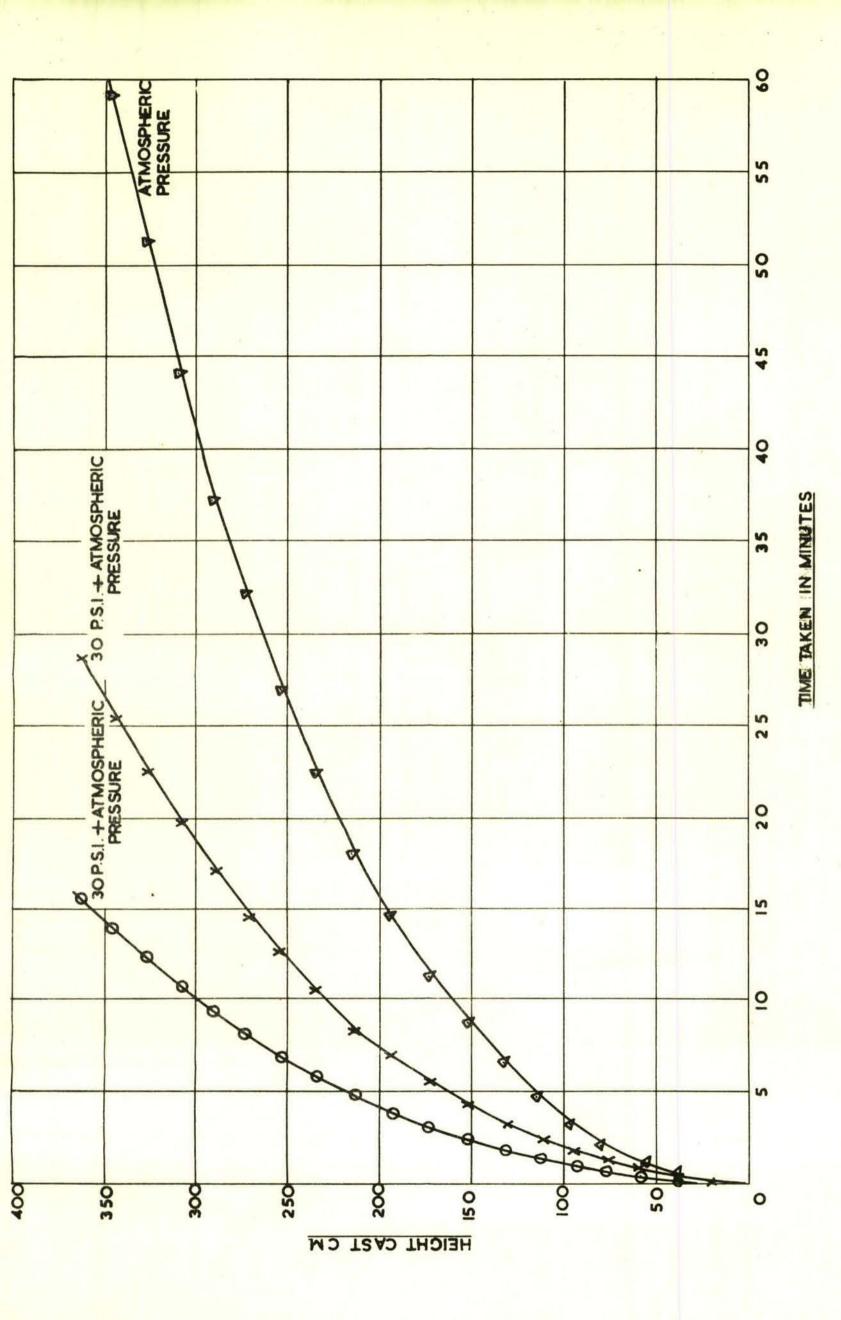
EFFECT ON THE VISCOSITY OF INCREASE IN CONCENTRATION OF SINGLE BASE-POWDER IN CASTING LIQUID

FIG



EFFECT OF HEIGHT OF FALL ON PACKING FRACTION

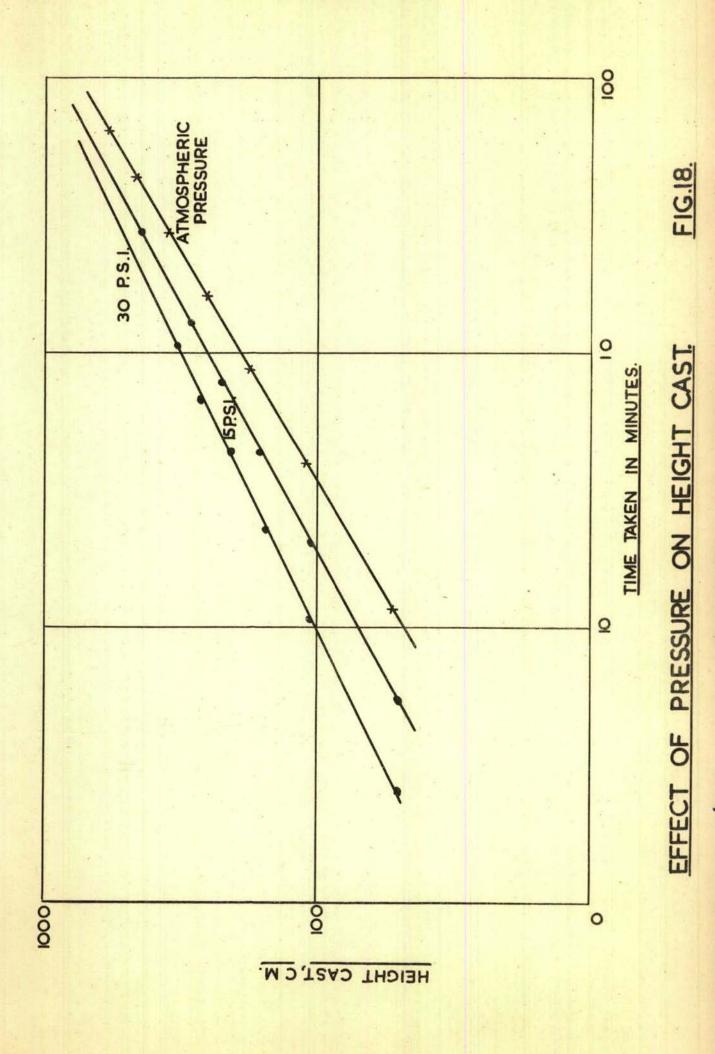
FIG. 16.



AS CASTING LIQUID. EFFECT OF PRESSURE ON HEIGHT CAST, 20°C, GLYCERIN & WATER

FIG . 17

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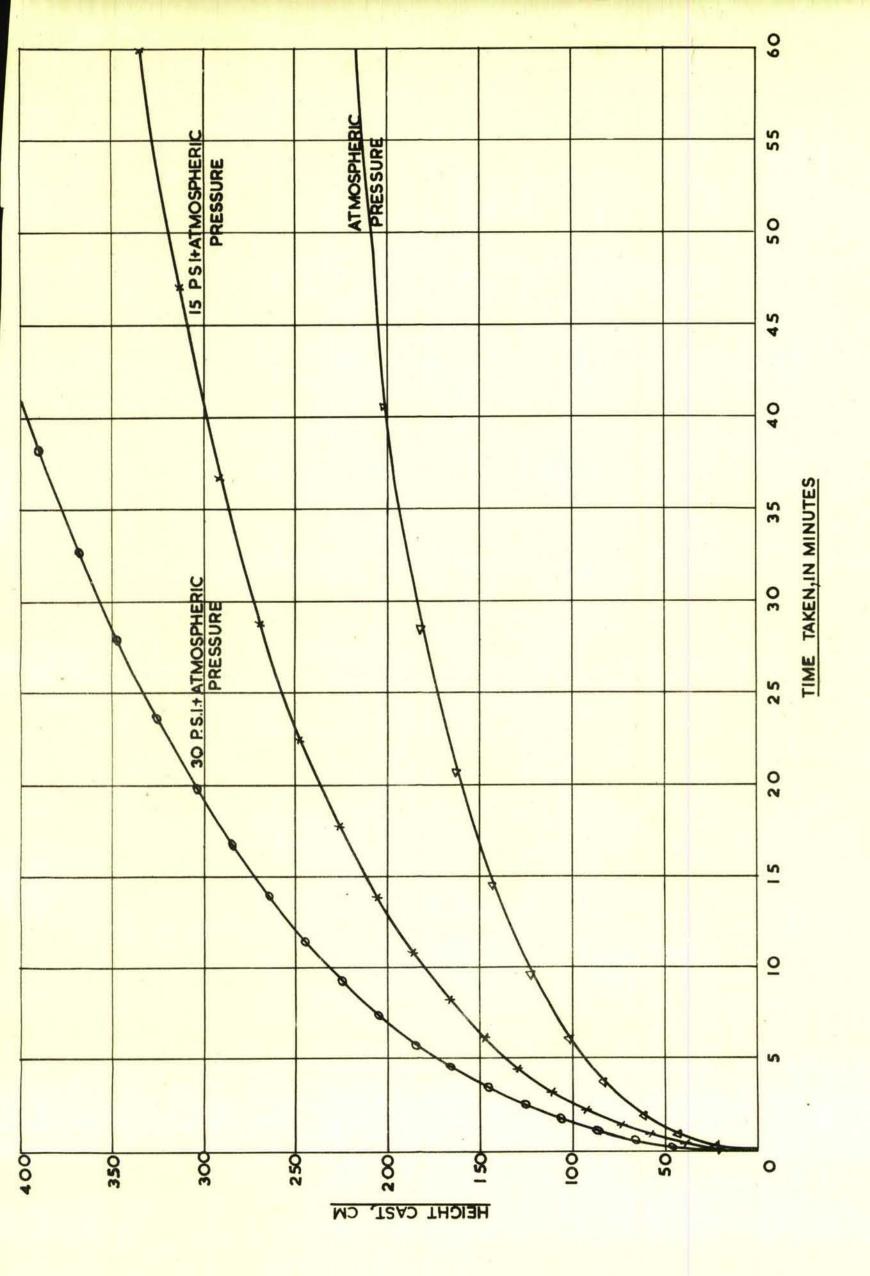
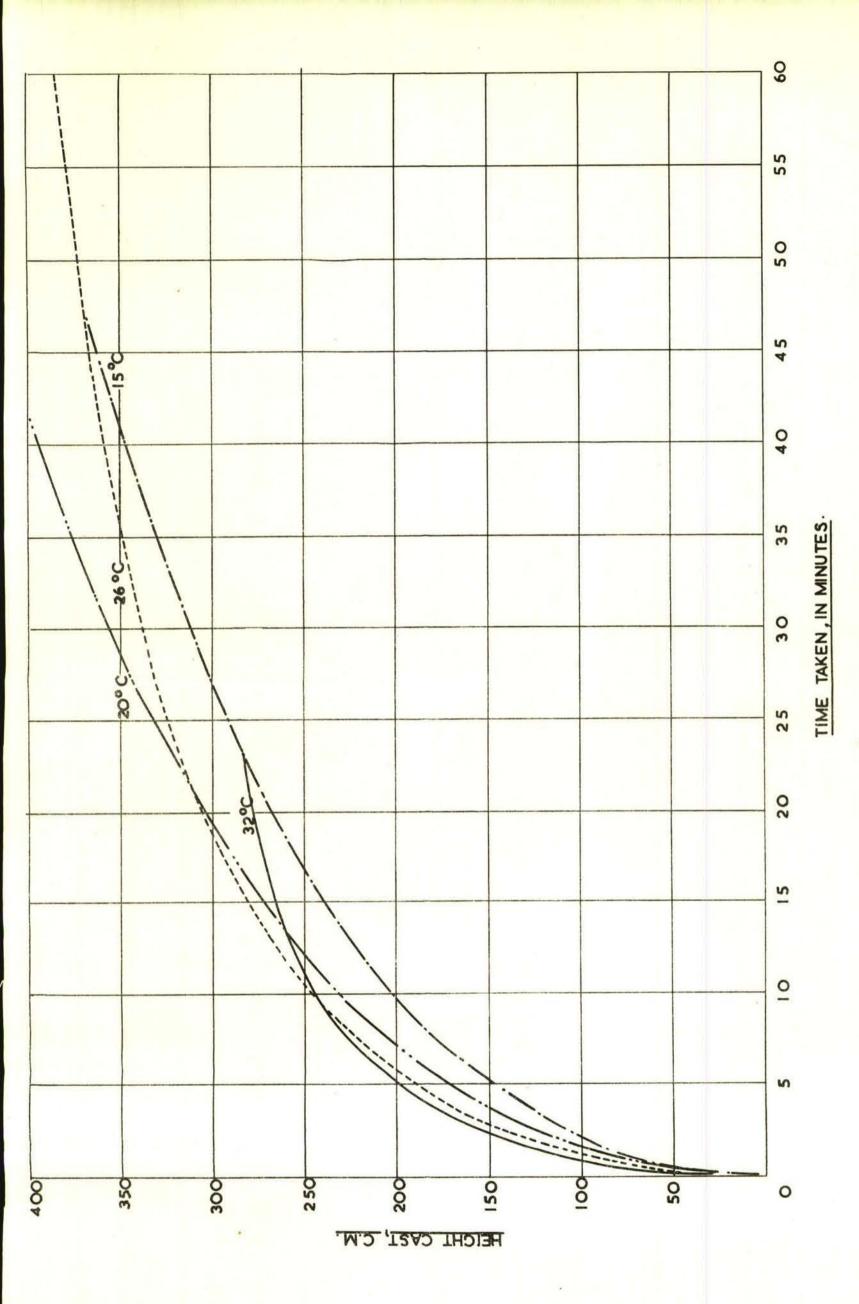


FIG. 19 PRESSURE ON HEIGHT CAST, 20°C, NITROGLYCERINE. P EFFECT



EFFECT OF TEMPERATURE ON HEIGHT CAST, 30 P.S.I. APPLIED NITROGEN PRESSURE.

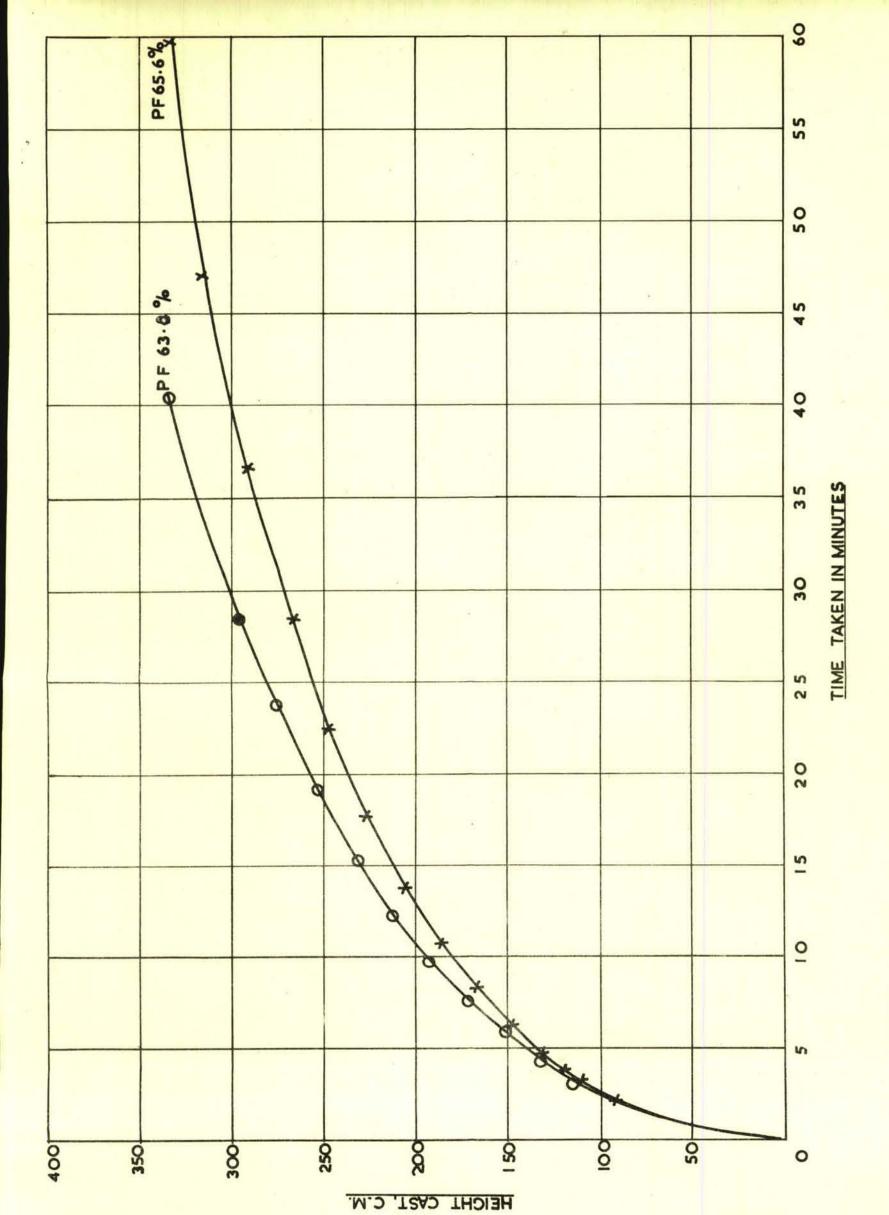


FIG. EFFECT OF PACKING ON HEIGHT CAST, 20° C, ISPS, APPLIED NITROGEN PRESSURE

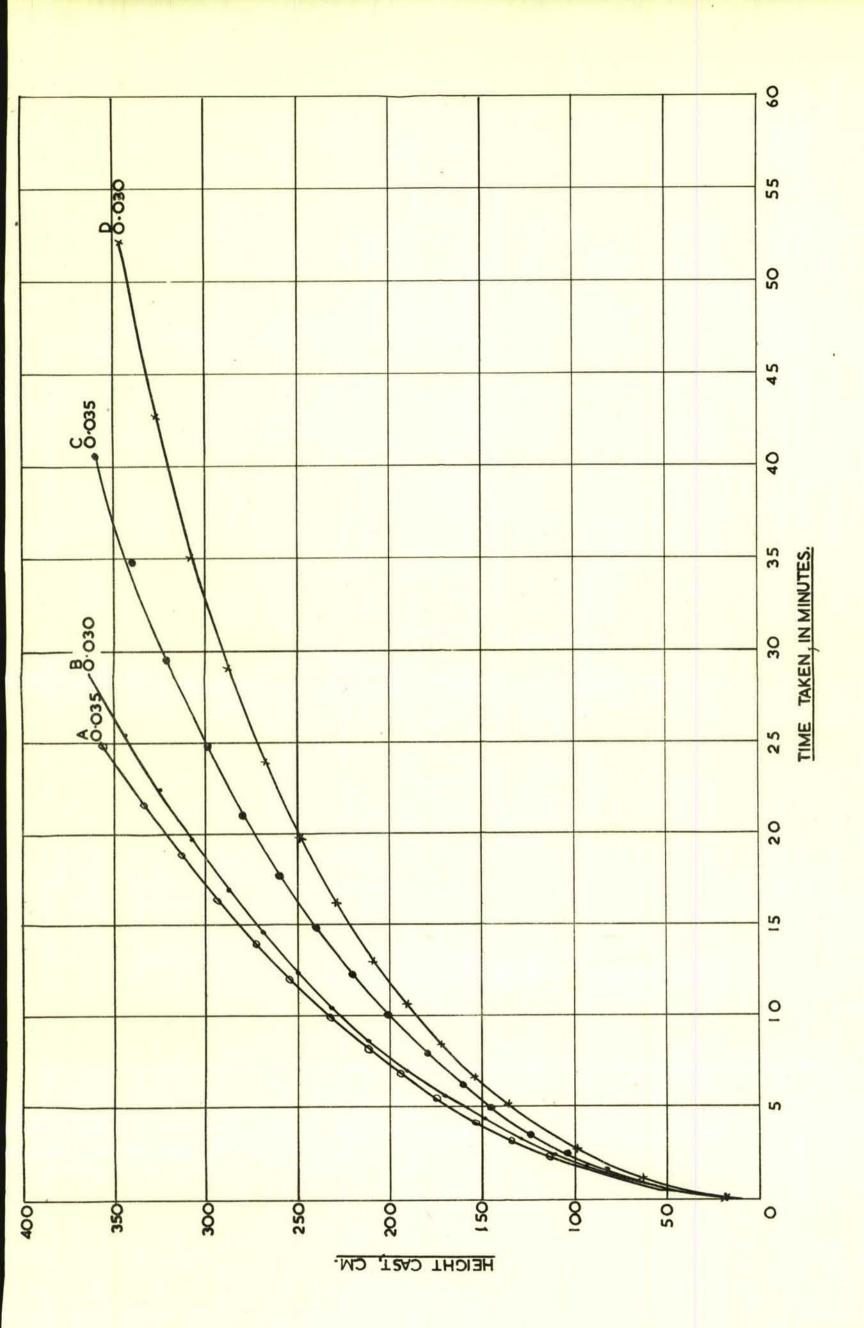


FIG. 22. EFFECT OF POWDER SIZE ON HEIGHT CAST, 20°C, IS P.S.I. APPLIED NITROGEN PRESSURE.

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